

What is claimed is:

- 1) A process for producing microsphere particles comprising the following steps:
  - a) charging to a first vessel water and a dispersion stabilizer and mixing to form a water phase;
  - 5       b) charging co-monomers to second vessel at 60 °C;
  - c) treating a charge control agent, a pigment and a micronized wax with a coupling agent;
  - d) charging to the second vessel the treated charge control agent, pigment and micronized wax of step (c);
  - 10       e) charging and dissolving the polymerization initiator to the second vessel to form a co-monomer phase;
  - f) pumping the co-monomer phase of step (e) through a disperser at a rate of about one liter per hour while simultaneously pumping the water phase of step (a) through the disperser at a rate of about  
15       four liters into a polymerization reactor;
  - g) mixing the water and monomer phases together in the polymerization reactor having a paddle rotation speed of about 200 rpm (30 m/sec) at a temperature of about 75 °C for about  
20       four to about six hours under an inert atmosphere to form a slurry of polymerized microsphere particles having a particle size from about 3 to about 20 microns;

- h) mixing the slurry of polymerized microsphere particles in the polymerization reactor under vacuum and a temperature of about 85 °C to remove any residual co-monomers;
  - i) acidifying the slurry to dissolve the dispersion stabilizer;
  - 5 j) passing the slurry of step (i) through a centrifuge to remove water and dispersion stabilizer dissolved therein to provide centrifuged polymerized microsphere particles; and
  - k) washing the centrifuged polymerized microsphere particles with water until a pH of 7 is achieved.
- 10 2) The process of claim 1 wherein the dispersion stabilizer is selected from the group consisting tricalcium phosphate, magnesium phosphate, zinc phosphate, aluminum phosphate, calcium carbonate, magnesium carbonate, calcium hydroxide, magnesium hydroxide, aluminum hydroxide, calcium metasilicate, calcium sulfate, barium sulfate, bentonite, silica, alumina, polyvinyl alcohol,
- 15 gelatin, methyl cellulose, methylhydroxypropyl cellulose, ethyl cellulose, sodium dodecylbenzene sulfonate and ethylene oxide addition products.
- 3) The process of claim 2 wherein the dispersion stabilizer is tricalcium phosphate.
- 20 4) The process of claim 1 wherein the co-monomers are selected from the group consisting of styrene, o-methylstyrene, m-methylstyrene, p-methylstyrene,  $\alpha$ -methylstyrene, p-chlorostyrene, 3,4-dichlorostyrene, p-phenylstyrene, p-ethylstyrene, 2,4-dimethylstyrene, p-tert-butylstyrene, p-n-hexylstyrene, p-n-

- octylstyrene, p-n-nonylstyrene, p-n-decylstyrene, p-n-dodecylstyrene, methacrylic acid ester derivatives such as methyl methacrylate, ethyl methacrylate, n-butyl methacrylate, isopropyl methacrylate, isobutyl methacrylate, t-butyl methacrylate, n-octyl methacrylate, 2-ethyl methacrylate, stearyl methacrylate, lauryl methacrylate, phenyl methacrylate, diethylaminoethyl methacrylate, dimethylaminoethyl methacrylate, methyl acrylate, ethyl acrylate, isopropyl acrylate, n-butyl acrylate, t-butylacrylate, isobutyl acrylate, n-octyl acrylate, 2-ethylhexyl acrylate, stearyl acrylate, lauryl acrylate, phenyl acrylate, ethylene, propylene, isobutylene, vinyl chloride, vinylidene chloride, vinyl bromide, vinyl fluoride, vinylidene fluoride, vinyl propionate, vinyl acetate, vinyl benzoate, vinyl methyl ether, vinyl ethyl ether, vinyl methyl ketone, vinyl ethyl ketone, vinyl hexyl ketone, N-vinylcarbazole, N-vinylindole, N-vinylpyrrolidone, vinyl naphthalene, vinylpyridine, acrylonitrile, methacrylonitrile and acryl amide.
- 5) The process of claim 5 wherein the co-monomers are styrene and n-butylacrylate.
- 6) The process of claim 1 wherein the charge control agent is selected from the group consisting of Bontron S-34, Bontron E-81, Bontron E-82, Bontron E-84, Bontron E-85, Bontron TRH, quarternary ammonium salts, nogrosine dyes, Bontron N-01, Bontron N-04 and Bontron N-07.
- 7) The process of claim 7 wherein the charge control agent is Bontron E-84.

8) The process of claim 1 wherein the pigment is selected from the group consisting of chrome yellow, cadmium yellow, hansa yellow G, condensed azo compounds, isoindolinone compounds, anthraquinone compounds, azo metal complexes, methine compounds, allylamide compounds, C.I. Pigment Yellow 12, C.I. Pigment Yellow 13, C.I. Pigment Yellow 14, C.I. Pigment Yellow 15, C.I. Pigment Yellow 17, C.I. Pigment Yellow 62, C.I. Pigment Yellow 74, C.I. Pigment Yellow 83, C.I. Pigment Yellow 93, C.I. Pigment Yellow 94, C.I. Pigment Yellow 95, C.I. Pigment Yellow 109, C.I. Pigment Yellow 110, C.I. Pigment Yellow 111, C.I. Pigment Yellow 128, C.I. Pigment Yellow 129, C.I. Pigment Yellow 147, C.I. Pigment Yellow 168, Toner Yellow 3 GP, brilliant carmine 6B, eosine, red iron oxide, phthalocyanine blue, Prussian blue, cobalt blue, carbon black, furnace black, magnetic materials, channel black, condensed azo compounds, diketopyrrolopyrrole compounds, anthraquinone compounds, quinacridon compounds, basic dye lake compounds, naphthol compounds, benzimidazolone compounds, thioindigo compounds, perylene compounds, C.I. Pigment Red 2, C.I. Pigment Red 3, C.I. Pigment Red 5, C.I. Pigment Red 6, C.I. Pigment Red 7, C.I. Pigment Red 23, C.I. Pigment Red 48:2, C.I. Pigment Red 48:3, C.I. Pigment Red 48:4, C.I. Pigment Red 57:1, C.I. Pigment Red 81:1, C.I. Pigment Red 122, C.I. Pigment Red 144, C.I. Pigment Red 146, C.I. Pigment Red 166, C.I. Pigment Red 169, C.I. Pigment Red 177, C.I. Pigment Red 184, C.I. Pigment Red 185, C.I. Pigment Red 202, C.I. Pigment Red 206, C.I. Pigment Red 220, C.I. Pigment Red 221, C.I. Pigment Red 254, Toner Magenta E 02, copper phthalocyanine compounds, anthraquinone compounds, C.I. Pigment blue 1, C.I.

Pigment blue 7, C.I. Pigment blue 15, C.I. Pigment blue 15:1, C.I. Pigment blue 15:2, C.I. Pigment blue 15:3, C.I. Pigment blue 15:4, C.I. Pigment blue 60, C.I. Pigment blue 62, C.I. Pigment blue 66 and Cyan BG.

- 5        9) The process of claim 1 wherein the polymerization initiator is selected from the group consisting of benzoyl peroxide, methyl ethyl ketone peroxide, diisopropyl peroxy carbonate, cumene hydroperoxide, t-butyl hydroperoxide, di-t-butyl peroxide, dicumyl peroxide, 2,4-dichlorobenzoyl peroxide, lauroyl peroxide, 2,2-bis-(4,4-t-butylperoxycyclohexane)propane and tris-(t-butylperoxy)triazine.

10

- 10) The process of claim 1 wherein the micronized wax has a melting point less than 70 °C.

- 11) The process of claim 1 wherein the coupling agent is a titanate coupling agent.

15

- 12) The process of claim 11 wherein the titanate coupling agent is selected from the group consisting of neopentyl(diallyl)oxy trineodecanonyl titanate (LICA 01), neopentyl(diallyl)oxy tri(dodecyl)benzene-sulfonyl titanate (LICA 09), neopentyl(diallyl)oxy tri(dioctyl)phosphato titanate (LICA 12),  
20        neopentyl(diallyl)oxy tri(dioctyl)pyro-phosphato titanate (LICA 38), neopentyl(diallyl)oxy tri(N-ethylenediamino) ethyl titanate (LICA 44), neopentyl(diallyl)oxy tri(m-amino)phenyl titanate (LICA 97), and neopentyl(diallyl)oxy trihydroxy caproyl titanate (LICA 99).

13) The process of claim 12 wherein the titanate coupling agent is  
neopentyl(diallyl)oxy tri(dioctyl)pyro-phosphato titanate (LICA 38).

5 14) The process of claim 1 further comprising the step of drying the centrifuged  
polymerized microsphere particles in a fluid bed dryer to provide dried  
polymerized microsphere particles.

15) The process of claim 14 further comprising the step of mixing the dried  
10 polymerized microsphere particles with fumed silica in a high-intensity mixer to  
obtain toner.

16) A process for producing microsphere particles comprising the following steps:

- 15 a. charging to a first vessel water and a dispersion stabilizer and mixing to  
form a water phase;
- b. charging co-monomers to second vessel at 60 degrees C;
- c. charging and dissolving the polymerization initiator to the second vessel to  
form a co-monomer phase;
- 20 d. pumping the co-monomer phase of step (c) through a disperser at a rate of  
about one liter per hour while simultaneously pumping the water phase of  
step (a) through the disperser at a rate of about four liters into a  
polymerization reactor;

- e. mixing the water and monomer phases together in the polymerization reactor having a paddle rotation speed of about 200 rpm (30 m/sec) at a temperature of about 75 °C for about four to about six hours under an inert atmosphere to form a slurry of polymerized microsphere particles having a particle size from about 3 to about 20 microns;
- f. mixing the slurry of polymerized microsphere particles in the polymerization reactor under vacuum and a temperature of about 85 °C to remove any residual co-monomers;
- g. acidifying the slurry to dissolve the dispersion stabilizer;
- h. passing the slurry of step (g) through a centrifuge to remove water and dispersion stabilizer dissolved therein to provide centrifuged polymerized microsphere particles; and
- i. washing the centrifuged polymerized microsphere particles with water until a pH of 7 is achieved.

15

17) The process of claim 16 further comprising the step of drying the centrifuged polymerized microsphere particles in a fluid bed dryer to provide dried microsphere particles.

20

18) A microsphere particle produced by the process of claim 1.

19) A color toner for use in a laser printer comprising the microsphere particle of claim 18.

20) A color toner for use in a photocopier comprising the microsphere particle of claim 18.